



GRANULAR MATERIAL MIXING:

Experiments for Calibration and Validation

Student Author



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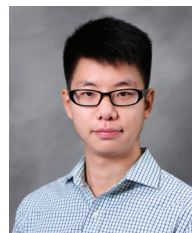
Mentors



Carl Wassgren is a professor of mechanical engineering and of industrial and physical pharmacy. His research interests focus primarily on modeling the dynamics of particulate and powder systems, with applications in mixing, segregation, coating, attrition, compaction, granulation, and characterization. In addition, he is interested in developing computational models for studying these

systems, including discrete element method, finite element method, and compartment and population balance models. This research has been applied to systems used

to process pharmaceuticals, food and consumer products, agrochemicals, and energy-related materials.



Yu Liu is a third-year PhD student working with Professor Carl Wassgren and Professor Marcial Gonzalez in the School of Mechanical Engineering. Before joining Purdue, he obtained his BA degrees in mechanical engineering from Shanghai Jiao Tong University

in China. His current research direction is focused on predicting granular material mixing and segregation using a finite element method and advection-diffusion-segregation multiscale model.



Marcial Gonzalez is an assistant professor of mechanical engineering. His research focuses primarily on predictive, multiscale modeling and simulation of microstructure evolution in confined granular systems, with an emphasis in manufacturing processes and the relationship between product fabrication and performance. Application areas of interest include: mixing, segregation, consolidation and compaction of powders, and performance of pharmaceutical solid products and energetic materials.

Abstract

Granular material mixing is common in many industrial applications ranging from the manufacturing of pharmaceuticals to the production of construction materials. Problems arise when proper blending is not achieved, which can lead to failure of the desired product. Currently most industries use trial and error to design blending processes. There is a clear need for not only quantitative, mechanics-based models for predicting blending performance but also experimental data that can be used for model calibration and validation.

In order to provide validation and calibration data in support of model development, two mixing experiments were designed and performed to measure the degree of mixing as a function of time: one in a rotating circular drum and the other in a Tote blender. The experimental apparatuses, operation, data collection and processing algorithms, and challenges encountered are described in this paper.

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Keywords

particle technology, mixing and blending, rotating drum, Tote blender, image analysis, segregation intensity

INTRODUCTION

Producing a homogenous blend of particles can be a critical operation. In the pharmaceutical industry, for example, the consequences can be severe if powder blends are not fully mixed (Fan, Chen, & Watson, 1970) since improper blending can lead to problems with efficacy and safety of the product.

Despite the importance of blending, virtually all powder blending processes are currently designed empirically due to the lack of quantitative, predictive tools applicable at industrial scales. Recently,

new a multiscale modeling tool has been proposed for predicting industrial blending (Liu, Gonzalez, & Wassgren, 2018), but there is a need for reliable experimental data that can be used to calibrate parameters in this model and also to provide experimental validation. This paper describes two different blending experiments designed to provide such data.

METHODS

Two blending systems are described in this section: a rotating drum and a Tote blender. The system geometries, materials used, operating procedures, and data collection and analysis methods are described.

Rotating Drum Experiment

A lab-scale rotating drum mixer was designed to calibrate one of the key parameters used in the multiscale model described by Liu et al. (2018). A photograph of the experiment is shown in Figure 1a. An acrylic circular drum of diameter of 150 mm and width of 50 mm was used to contain the material. The drum was made transparent so that material near the front face of the cylinder could be imaged. The backside of the drum was built to be detachable to allow for filling and discharging of the drum. Grip tape was used to seal the backside of the drum and provide extra friction for the drive shaft. A motor rotated the drive shaft that in turn rotated the drum. Rubber bands covered the drive shafts to prevent slipping. The rotation speed was set to 3.3 rpm, which ensured that no slip would occur between the material and the drum. Under these conditions, the material moved in a rolling regime (Mellmann, 2001), where material continuously avalanches down the free surface of the bed.

The drum was partially filled with a 50/50 mixture of 1 mm diameter red and blue coated glass spheres (Fire Mountain Gems and Beads). The spheres were identical, except for color. Glass spheres are used frequently as a standard test material since they are easily characterized, cohesionless, and easy to procure, and flow well. The bulk density, internal friction angle, and wall friction coefficient of the material were measured using a Schulze Ring Shear Tester (Model RST-XS) and are summarized in Table 1, along with other significant experiment parameters. The fill level, defined here as the maximum level-surface height of the particle bed to the diameter of the drum, was 32%. Initially the spheres were separated side-by-side in the drum (Figure 1b).

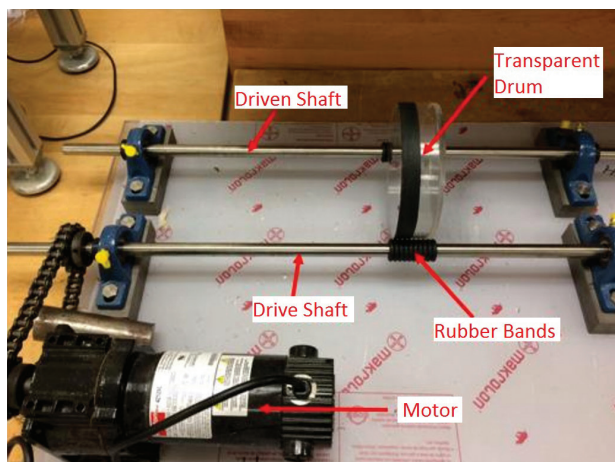


Figure 1. (a) The rotating drum experiment setup; (b) Initial filled state of the rotating drum with red and blue spheres separated.

To gather the images required to analyze the degree of mixing, a Samsung Galaxy S6 camera was positioned to view material at the front face of the drum. The camera frame rate was 60 frames per second with an image resolution of 1920 x 1080 pixels. The frame rate and resolution were sufficient to capture the movement of the spheres without streaking artifacts. Lighting placement played an important part in reducing glare and light reflection. Two light sources with diffusing shades were located approximately three feet from and to either side of the drum.

Individual frames from the recorded video were analyzed using an in-house MATLAB code in order to determine the degree of blending between the red and blue spheres. A summary of the image processing steps is shown in Figure 2. For each image, the material domains (red and blue glass spheres)

Parameter	Value
Inner drum diameter (mm)	150
Inner drum width (mm)	50
Glass sphere diameter (mm)	1
Filling level (% of max level depth)	32
Wall friction coefficient	0.324
Internal friction angle (degree)	27.1
Material bulk density (kg/m ³)	1500
Drum rotation speed (rpm)	3.26

Table 1. The rotating drum experiment parameters.

were extracted using the freeware program ImageJ (Schneider, Rasband, & Eliceiri, 2012), which was used to identify the glass spheres from a white background. The RGB image was further analyzed to extract only the red component of each image's pixels, with a red pixel color value ranging between 0 (no red) and 255 (all red). A threshold value of 80 was selected to differentiate between the red and blue pixels, with a value larger than 80 indicating that a pixel was red while a value smaller than 80 indicated that the pixel was blue. This threshold value was chosen to ensure that at any point in time the fraction of red pixels in the entire system was $50 \pm 5\%$ since the system consisted of 50% red spheres.

After distinguishing red and blue pixels, a black and white binary image was generated, with black corresponding to the blue pixels and white corresponding to the red ones. Note that since spherical glass beads were used in the experiment, reflections and shadows were introduced due to the light source. An image correction algorithm was developed in the MATLAB program in an attempt to account for these effects. The algorithm checked the binary image for small

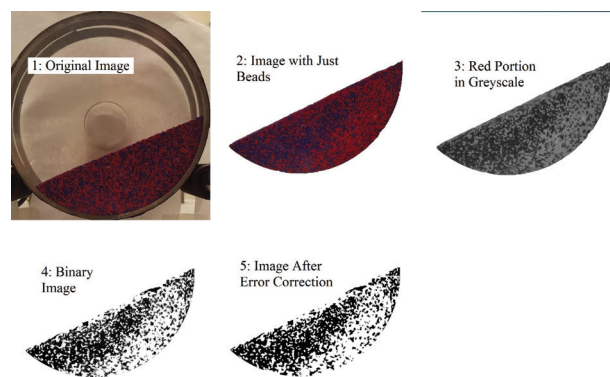


Figure 2. The image analysis algorithm steps for a single image. The steps proceed from 1 to 5.

central patches of pixels that were surrounded by pixels of the opposite type. If found, the pixel values of these central patches were flipped to “fill in” the particle. This method worked well since the light reflections and shadows were small compared to the sphere sizes.

The final binary image was used to compute the segregation intensity I for the system at that time instant. Segregation intensity is a standard method for quantifying the degree of blending of particulate systems and is defined as the variance in the spatial distribution of the concentration of a given particle species, in this case the red spheres, to the variance that would occur if the system was fully segregated, i.e., completely unmixed. Thus, a value of $I = 0$ corresponds to a perfectly mixed system while $I = 1$ when the system is fully segregated. Further details on the segregation intensity can be found in Liu et al. (2018).

In calculating the spatial distribution of red particle concentration, each video image was divided into a collection of nonoverlapping, square grid cells. The cell size varied from three to 10 sphere diameters on a side. The number of red pixels in each cell was divided by the total number of pixels in the cell to determine the red particle concentration. To avoid including only partially filled or empty cells, at the system boundaries for example, only those cells that contained at least 95% material by area were included in the calculations.

Tote Blender Experiment

The second blender system investigated in this work was a lab-scale Tote blender, as shown in Figure 3. The data gathered from this experiment was intended for model validation. The Tote blender in this experiment had a rotational speed of 7.5 rpm. The material used in this experiment consisted of 1 mm transparent and blue glass spheres, which had properties identical to those used in the rotating drum experiments described previously (other than color). The blender was filled to 40% of its height, with the transparent and blue glass spheres initially separated side-by-side (refer to Figure 4).

Unlike the rotating drum, particles in the Tote blender were not visible during operation of the blender. Thus, the video recording method for determining the blending performance could not be used. Instead, a thief probe method for collecting samples was attempted. A thief probe (Figure 5) consists of a cylinder within a cylinder, with an opening cut into

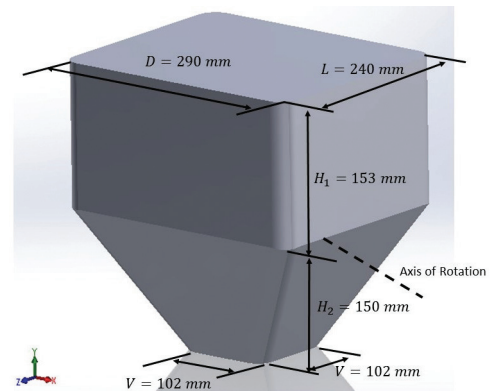


Figure 3. (a) The lab-scale Tote blender used in the current work; (b) A dimensioned schematic of the blender.

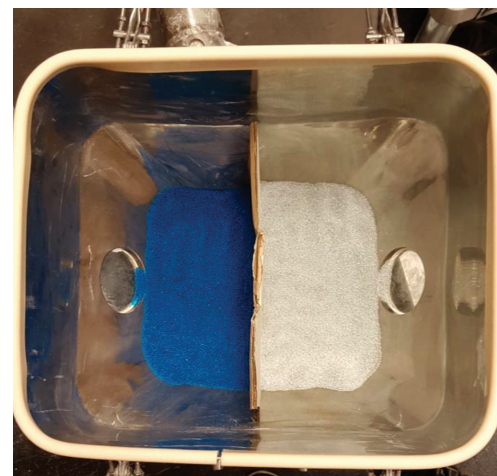


Figure 4. Initial condition in the Tote blender experiment with the blue and transparent glass spheres separated laterally.

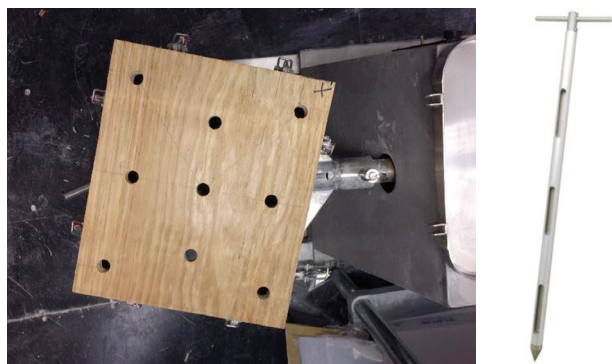


Figure 5. (a) The sample template; (b) The thief probe used in the current work.

each cylinder's side. When inserted into the particle bed, the openings are misaligned so no particles enter the inner cylinder chamber. When the desired location is reached, the inner cylinder is rotated so that the outer and inner openings align and particles fall into the inner chamber. The inner cylinder is rotated again once the sample has been collected and the thief probe is removed from the particle bed. Using this device, samples of material may be collected within the bulk of the material.

In order to collect particle samples and evaluate the degree of blending as a function of time, the Tote blender had to be stopped after each revolution. Nine samples were collected from the same locations within the blender with the aid of a wooden template (Figure 5). The samples were collected at various lateral and vertical locations. The sampling process was repeated eight times, resulting in a total of 72 collected samples. Each sample volume was approximately 2 ml, resulting in approximately 2,000 glass spheres in each sample. The samples were not returned to the blender after collection, but the total amount of collected material, approximately 144 ml, was small in comparison to the total amount of material initially in the blender, 2800 ml.

An image analysis technique was used to determine the number concentration of transparent spheres in each sample. The technique used in this experiment is different from the one described for the rotating drum experiment, as the use of transparent spheres resulted in reflections from the spheres that caused the previously described image processing algorithm to fail. For the Tote blender experiment, each thief probe sample was poured into a small glass container such that a monolayer of spheres was produced. The glass container was supported approximately 0.3 m above a white background with a black rectangle located at the center. When

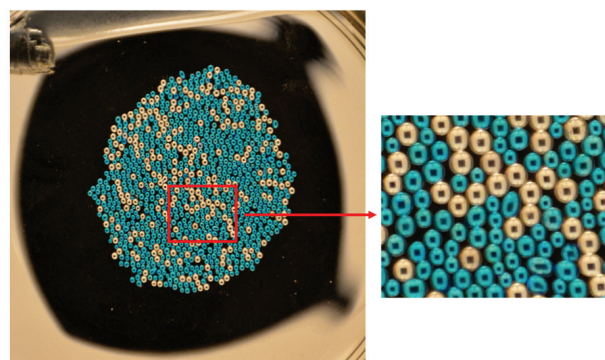


Figure 6. The finalized refraction image with close-up.

imaged from above, an image of the black rectangle was clearly observed in the center of each particle, making it straightforward to identify individual particles (Figure 6).

The ImageJ software package was used to analyze the resulting sample images. An algorithm, summarized in Figure 7, was designed to count the number of spheres in each sample. First, each image was split into RGB components, with each component's pixel value ranging from 0 to 255. Second, a threshold value of 160 for the red component value was selected and the original pictures were converted into black and white binary images based on this threshold value. Third, an ImageJ plugin called Analyze Particles was implemented. This plugin is used to detect and count the number refracted shapes, i.e., glass spheres, within the image. Using the resulting transparent particle concentration data from the samples, a segregation index value was calculated.

To ensure that the image processing algorithm was working properly, three of 72 samples were randomly chosen, and the numbers of blue and transparent spheres were counted manually. The image processing values matched the manual count values to within approximately 1% (Table 2).

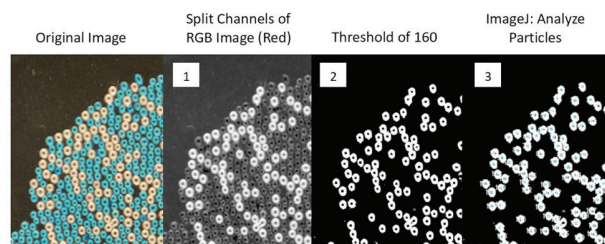


Figure 7. The image analysis algorithm for a single image close-up. The steps proceed from 1 to 3.

Sample number	Number of white beads			Number of all beads		
	Manual	ImageJ	Error (%)	Manual	ImageJ	Error (%)
1	1272	1286	1.09	1521	1523	0.13
2	395	396	0.25	2075	2069	0.29
3	962	971	0.93	1912	1919	0.37

Table 2. Comparison of the image processing and manual sphere counts.

RESULTS

The results from the two blending experiments are presented in the following subsections. The rotating drum experiment data is used for model calibration while the Tote blender is used for model validation.

Rotating Drum Experiment

Several snapshots of the rotating drum system are shown in Figure 8 for different numbers of drum revolutions. Qualitatively, the mixing dynamics in the experiment followed the same trends as reported in previous work (Hajra and Khakhar, 2005). The mixing consists of a combination of advective mixing in which bulk movement of the material occurs, and diffusive mixing, which occurs locally due to random particle movement. Although not easily visible in the individual images shown in Figure 8, the flow field consists of a thin active region at the bed's free surface where material is subject to shear, and a passive region below the active region where material moves in solid body rotation. Mixing in the system occurs primarily in the active region.

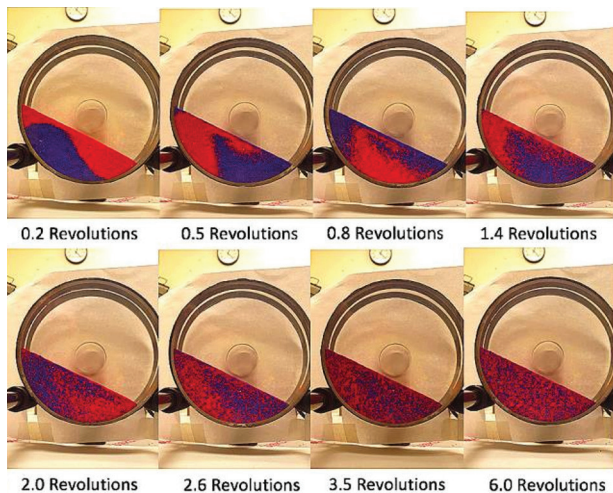


Figure 8. Images showing the state of particle mixing for different numbers of drum revolutions in the rotating drum experiment.

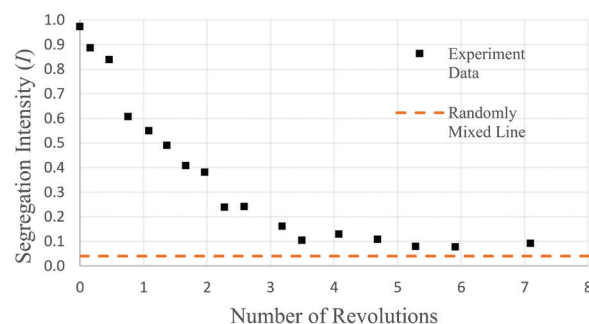


Figure 9. Segregation intensity I plotted as a function of the number of drum revolutions for the rotating drum experiment. The horizontal dashed line is the predicted randomly mixed state for a sampling bin size of five particle diameters.

The segregation intensity I is plotted in Figure 9 as a function of the number of drum revolutions (black squares in the figure). The sampling cell size for this case was five particle diameters. The segregation intensity starts at a value near 1, which implies a nearly perfectly segregated system. As the number of drum revolutions increases, the segregation intensity decreases, indicating blending of the red and blue particles. After approximately four drum revolutions the segregation intensity remains nearly constant at a value of approximately 0.1. It can be shown that the smallest segregation intensity for a system involving random mixing corresponds to a nonzero value that depends on the number of particles in the sample (Danckwerts, 1952). For the case shown in Figure 9, the randomly mixed segregation intensity is 0.04, which is nearly the measured value.

Tote Blender Experiment

The segregation intensity is plotted as a function of the number of blender revolutions for the Tote blender in Figure 10. As with the rotating drum experiment, the segregation index has a value near 1 at the start of the experiment since the system is segregated. However, the segregation index in the Tote blender decreases more rapidly than the rotating

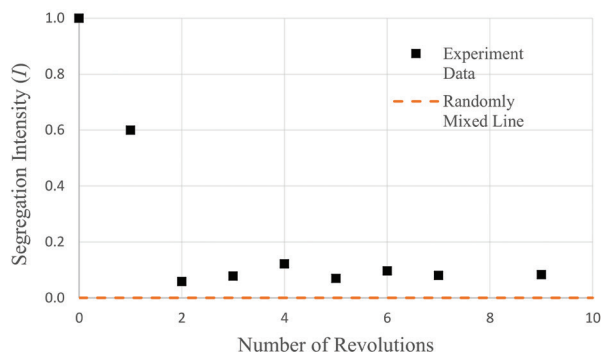


Figure 10. Segregation intensity plotted as a function of the number of blender revolutions for the Tote blender experiment.

drum and reaches a steady mixed state, with a segregation index value of approximately 0.08 within two drum revolutions. The randomly mixed segregation intensity value is 0.0005, which is much smaller than the measured value. Since the measured segregation intensity appears to have reached an asymptotic value that is two orders of magnitude larger than the randomly mixed state, it suggests that either the system may have one or more “dead zones” at the sampling locations. In fact, a close examination of Figure 11 shows that presence of a dead zone at the upper left of the figure where transparent spheres remain mostly unmixed.

There are three reasons for such a rapid change in the segregation index during the first few blender revolutions. First, the Tote blender is a more effective blender than a rotating drum. The orientation of the Tote blender’s rotation axis is offset from the

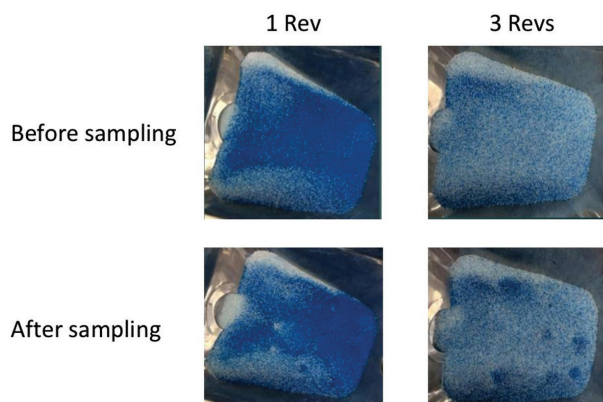


Figure 11. Perturbations to the bed caused by thief probe sampling. The top photographs show the bed prior to sampling and the bottom images show the bed after sampling. Regions where the thief probe entered the bed can be clearly observed. A mixing “dead zone” is observed in the upper left portion of the images.

blender’s geometric line of symmetry, which produces more rapid, three dimensional mixing than the planar mixing in a rotating drum. Second, starting and stopping the blender likely causes additional blending as material is subject to sudden accelerations and decelerations. Third, the thief probe sampling method causes significant perturbation of the bed and also contributes to mixing. As the thief probe is pushed into the bed, it drags particles from the upper layers down to the lower layers. Similarly, when the probe is removed, particles are dragged upwards. This effect has been observed by Muzzio, Robinson, Wightman, and Brone (1997). With nine samples collected after each blender revolution, these perturbations cause considerable mixing between the upper and lower portions of the bed. These effects can be observed in Figure 11. The rapid mixing and thief probe perturbations make the current Tote blender experiments less useful and reliable to compare to model predictions.

CONCLUSION

This paper presents the design, operation, results, and analysis of particle mixing in rotating drum and Tote blenders. The purpose of these experiments was to provide experimental data for use in model calibration and validation. A video recording and image analysis technique was used to gather particle concentration data in the rotating drum experiment, and a thief probe collection method and subsequent image analysis technique was used in the Tote blender experiments. Mixing data from the rotating drum behaved as expected, with the segregation intensity beginning near a value of 1 and decreasing over approximately four drum revolutions to a randomly mixed state. The Tote blender experiment, however, produced mixing so rapidly that the sampling rate (once per blender revolution) was too coarse to provide good temporal resolution of the mixing. In addition, the perturbations to the bed caused by thief probe sampling significantly enhanced mixing drawing into question the reliability of the measured segregation intensity values. Despite the rapid decrease in the “segregation index,” a small mixing dead zone was also observed in a portion of the blender, which likely resulted in the randomly mixed segregation index value not being reached. Future Tote blender experiments could be improved by only sampling once during the experiment rather than after every blender rotation. This approach would improve the data reliability but greatly increase the complexity of the experiment since it would need to be restarted from the initially segregated state after every sample.

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